



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3067

Toxaphene in Methanol

This Standard Reference Material (SRM) is a solution of technical toxaphene (Chemical Abstracts Registry Number 8001-35-2) in methanol. This SRM is intended primarily for calibrating chromatographic instrumentation used for the determination of technical toxaphene. Because of its miscibility with water, SRM 3067 can also be used to fortify aqueous samples with known amounts of toxaphene. A unit of SRM 3067 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Toxaphene: The certified concentration [1,2], given below, is based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Toxaphene 26.1 mg/kg \pm 1.0 mg/kg or 20.9 mg/L \pm 0.8 mg/L

The results are expressed as the certified value \pm the expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and gas chromatographic measurements. The expanded uncertainty, at the 95 % level of confidence, is calculated as $U = kc_c$, where u_c is a combined standard uncertainty calculated according to the ISO Guide [3] and $k = 2$ is the coverage factor. The value of u_c includes an allowance for differences between the concentration determined by gas chromatographic measurements for various sources of toxaphene and gravimetric preparation. The concentration expressed as a volume fraction (in mg/L) was obtained by multiplying the certified value, expressed as a mass fraction, by the measured density (22 °C) of the SRM solution, 0.800 (0.016) g/mL where 0.016 represents one standard deviation (1s) and is incorporated in the volume fraction uncertainty.

Expiration of Certification: The certification of this SRM lot is valid until **31 March 2013**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The coordination of the technical measurements leading to the certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Analytical measurements of the SRM were performed by M.M. Schantz and S.J. Broadwater of the NIST Analytical Chemistry Division.

The support aspects involved in the issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 May 2003

John Rumble, Jr., Chief
Measurement Services Division

Preparation of the SRM was performed by M.P. Cronise of the NIST Measurement Services Division and M.M. Schantz of the NIST Analytical Chemistry Division.

Partial support for the preparation and certification of this SRM was provided by the U.S. Environmental Protection Agency Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

NOTICE AND WARNING TO USERS

Handling: This material contains toxaphene, and should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

INSTRUCTIONS FOR USE

Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified value to be valid within the stated uncertainty. Because of the volatility of methanol, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS¹

The toxaphene used in the preparation of this SRM was obtained from the former U.S. EPA Repository, Research Triangle Park, NC. The solution was prepared at NIST by weighing and mixing the toxaphene into the methanol. The weighed toxaphene was added to the methanol and mixed until completely dissolved and homogenized. The total mass of this solution was measured, and 1.2 mL aliquots were dispensed into 2-mL amber glass ampoules, which were then flame sealed.

Aliquots from two sets of nine ampoules, selected randomly, were analyzed in duplicate using capillary gas chromatography with electron capture detection and an immobilized non-polar stationary phase column. An internal standard solution containing hexachlorobenzene and PCB 209 was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of toxaphene (from the same source as used to prepare SRM 3067 for the first set of nine ampoules and from four different sources for the second set of nine ampoules) and internal standard compounds in methanol were chromatographically analyzed to determine response factors for dominant toxaphene peaks relative to the internal standards (see Figure 1). This approach is similar to EPA Method 505 (Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products in Water by Microextraction and Gas Chromatography, revision 2.0).

¹Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

- [1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).
- [2] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, U.S. Government Printing Office: Washington, DC (1995).
- [3] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.

